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DEVELOPMENT OF A FABRICATION PROCESS FOR LARGE CYLINDERS FROM MIXTURES OF SPHERICAL POWDERS OF HIGH STRENGTH ALUMINUM AND MAGNESIUM ALLOYS 7

August 1971

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FINAL REPORT FOR PROGRAM III

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Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER Watertown, Massachusetts 02172

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DEVELOPMENT OF A FABRICATION PROCESS FOR LARGE CYLINDERS FROM MIXTURES OF SPHERICAL POWDERS OF HIGH STRENGTH ALUMINUM AND MAGNESIUM ALLOYS

Technical Report by

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August 1971

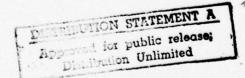
Final Report for Program III

Contract Number DAAG46-71-C-0076

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#### **ABSTRACT**

Two fabrication processes for the production of cylindrical shells approximately 6-inch 0.D. x 1/2-inch wall made from mixtures of high strength aluminum alloy and magnesium alloy powders were investigated. The first method consisted of a sequence of consolidation, upsetting and back extrusion. This approach did not yield a sound product. The second method consisted of the direct extrusion of a heavy wall tube. This approach was shown to be feasible and to produce sound high strength components. Reduced scale extrusions of rods and tubes were carried out and led to the design of a full scale fabrication process.



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### I. INTRODUCTION

In work performed previously for the Army Materials & Mechanical Research Center  $(Refs\ l\ -\ 4)$  it has been shown that shapes made of high strength alloys of magnesium and aluminum alloy powders and consolidated by extrusion have properties exceeding those obtained in cast and wrought alloys of the same composition. It has also been shown that composites consisting of two different powders combine some of the more desirable properties of each.  $(Ref\ 5)$ 

A method of consolidation by extrusion of mixtures of the high strength aluminum alloy 7075\* and the high strength magnesium alloy ZK60A\*\* was demonstrated in earlier work. (Ref 4) It was the objective of the work reported here to adapt this technique to the production of large (approximately 6-inch 0.D.) cylinders for use in ordnance applications.

#### II. OBJECTIVE

The objective of this work was to develop a fabrication process for cylindrical bodies, 6-inch 0.D. x 1/2-inch wall x 6 inches long, from two mixtures of aluminum and magnesium alloys, one consisting of  $70^{\text{V}}/\text{o}$  7075-  $30^{\text{V}}/\text{o}$  ZK60A and one consisting of  $70^{\text{V}}/\text{o}$  ZK60A and  $30^{\text{V}}/\text{o}$  7075 alloy powders.

<sup>\* 7075 = 1.6%</sup>Cu, 5.6%Zn, 2.5%Mg, 0.7% max. Fe.

<sup>\*\*</sup> ZK60A = 4.5%Zn, 0.45%Zr.

The objective of the initial small scale work was to demonstrate a process combining upset forging and back extrusion. If this process had been successfull, the full size cylinders could have been produced on relatively small equipment. When it became clear that this process could not yield sound cylinders, the objectives were modified and the work was aimed at demonstrating, on a reduced scale, the feasibility of producing cylinders by direct extrusion.

#### III. TECHNICAL APPROACH

In work carried out previously, it has been shown that the Al-Mg alloy powder mixtures could be extruded successfully at temperatures of 250°F and above. Material extruded at these temperatures lacked response to heat treatment. Attempts to extrude the material at room temperature, 145°F and 190°F, did not yield sound shapes. A temperature of 212°F (boiling water) appeared to permit the production of sound shapes without extensive loss of aging response in the magnesium alloy. Therefore, an extrusion temperature of 212°F was used in the present work. At this temperature the powder mixtures are still very stiff and the reduction in area which can be obtained in one step is limited by the permissible tool stresses as well as by the available capacity of the extrusion and forging equipment. In previous work, it had been shown that a reduction in area in extrusion of 10 times would yield sound rods. A 10 times reduction in area could not be obtained in the proposed compacting-upsetting-cupping sequence. It was also not known if the powders would respond to several successive working steps in the same manner in which they would respond to a single operation having an equivalent amount of total deformation.

The proposed working sequence is shown schematically in Figure 1. It consisted of three separate steps: (1) <u>Compacting</u> of powders within an aluminum container between hardened steel penetrators at room temperature, (2) <u>Hot Upsetting</u> the compact to a larger diameter "pancake" and (3) <u>Cupping</u> or back extrusion of the pancake to form a closed cylinder.

When the above approach did not yield sound usable cylinders, the technical approach was changed toward direct extrusion of the cylinders illustrated in Figure 2. It was felt that the aluminum canning technique, used in earlier extrusion work as well as in the cupping technique used in this program, would not be suitable for large scale direct extrusion. Therefore, a copper canning technique was developed first through extrusion of solid copper canned rods and then through the reduced scale extrusion of a heavy wall tube. The data obtained permitted the design of the large billets to be used in the production of the full scale cylinders.

#### IV. WORK DESCRIPTION

## A. Production of Alloy Powders

The powders used in this program were produced by the REP<sup>TM</sup> method at the Nuclear Metals Division of Whittaker Corporation. In this process, Figure 3, electrodes of the alloy 2-1/2 inches in diameter are rotated at high speed around their longitudinal axis within a chamber containing a helium atmosphere. The end of the rotating electrode is melted by an electric arc. The liquid metal flying off the rotating electrode forms a fine spherical powder which solidifies before contacting the walls of the chamber.

Approximately 60 pounds of -35 mesh 7075 alloy powder and 21 pounds of -35 mesh ZK60A alloy powder was produced for this program. (Powder characterization and chemical certificates are shown in appendices A & B.) The ZK60A powder was stored in a refrigerator at a temperature of 45°F when not in use.

## B. Mixing of Powders

The specific compositions which were to be produced through the cupping process were  $70^{V}/oA1-30^{V}/oMg$  and  $30^{V}/oA1-70^{V}/oMg$ . This corresponds to  $78^{W}/oA1$ ,  $22^{W}/oMg$  and to  $40^{W}/oA1-60^{W}/oMg$ . Through an error in computation, the mixtures used in the cupping work were actually  $60.4^{V}/oA1-39.6^{V}/oMg$  and  $21.9^{V}/oA1-78.1^{V}/oMg$  ( $70^{W}/oA1$ ,  $30^{W}/oMg$  and  $30^{W}/oA1$ ,  $70^{W}/oMg$ ). The AMMRC Technical Project Manager was advised of the error and stated that these compositions would be satisfactory for the experimental phases of this study.

This error was corrected after the first 10 billets for compacting had been prepared.

All powders were mixed for 3-5 minutes in a V-cone blender with intensifier. Enough powder for each billet was blended separately. The mixed powders were carefully spooned into the billet in order to avoid segregation.

## C. Fabrication of Ten Cups and Pancakes

In order to facilitate an understanding of the fabrication history of each of the cups and pancakes, the fabrication steps are indicated schematically in Figure 4.

## 1. Billet Preparation

A total of 10 cans of 6061 aluminum alloy tubing 3-1/2 inch 0.D., 3-inch 0.D., 6-3/4 inches long were prepared. The 6061 aluminum tubing was heat treated for maximum strength by heating for 15 minutes to 900°F and water quenching. An 1100 aluminum end plug 1/4-inch thick was pressed into one end of the can. The powder mixtures were then placed into the can. The pour density of spherical powder is approximately 62%. A second 1100 aluminum end plug was then pressed into the end of the can on top of the powders.

## 2. Compacting

All billets (#1 through #10) were compacted at room temperature, using double steel penetrators 3 inches diameter, in an extrusion liner having a bore of 3.545 inches. The compacting force was 900 tons (92 tsi).

#### 3. Upsetting

Billets 1, 2, 3, 4 and 6, 7, 8 and 9 were processed by upsetting at elevated temperature.\* The compacted cans were trimmed of excess canning material. Aluminum discs 4-1/2 inch 0.D. x 1/8-inch thick, were tack welded to each end of the compacts in order to center the compacts in the liner. All billets were heated for 5-10 minutes in boiling water. Billets #1, 2, 6 and 7 were upset directly in an extrusion liner preheated to 212°F. Billets #3, 4, 8 and 9 after heating in water were

<sup>\*</sup>Upsetting from 3-1/2 inch diameter to 4-1/2 inch diameter results in an increase in area of 65.5%.

placed in the extrusion liner preheated to 250°F for 10 minutes. The extrusion liner diameter was 4.555 inches and the upset force was 1000 tons (62 tsi). All billets were removed from the extrusion liner as fast as possible and quenched in cold water.

## 4. Cupping

Billets #2, 3, 5, 7, 8 and 10 were back extruded into cups. The billets which had been previously upset in the 4.545-inch liner were machined to 4.500 inches to fit again into this liner for cupping. The punch used for the cupping had a diameter of 2.750 inches and a length of 4-1/4 inches with 1/2 inch and was made from hardened H21 tool steel. It was centered in the liner with carbon steel slip rings 4.5-inch 0.D. x 2.750-inch I.D. Billets #2 and 7 were cupped at 212°F (boiling water), billets #3, 5 and 8 were cupped at 250°F. The method of heating was identical to that described for the upsetting above. The cupping force was 1100°F (68 tsi).

#### 5. Evaluation

As part of the program, an extensive evaluation of the cups had been planned. When the cups and the upset pancakes were sectioned, it was evident that the quality of all the pieces was so poor that this approach to producing sound cylinders was unlikely to be successful. Figures 5 through 8 show some of the cups and pancakes in cross section. In most cases the powder particles were poorly bonded and the geometry of the cups was highly irregular.

At a meeting with the AMMRC technical monitor, it was decided to stop all further efforts aimed at cupping, including the planned evaluation and to concentrate the remaining funds on the development of the direct extrusion method.

#### D. Direct Extrusion

In order to use the direct extrusion approach a large extrusion or forging press and large extrusion tooling will be required. Such a press and tooling is available to AMMRC at the Wyman-Gordon plant, Millbury, Massachusetts. The tooling which had been used by AMMRC for another project consists of a container with an I.D. of 12.88 inches, a die having an aperture of 6.62 inches and a mandrel 4.5-inch 0.D. This tooling will result in an extrusion reduction R of 6:1 (R = cross sectional area of billet/cross sectional area of extruded pipe). The experimental work carried out under the present program was aimed at demonstrating that this tooling would produce a sound cylinder having attractive physical properties. The work consisted of the extrusion of a number of solid rods under various conditions, and the extrusion of reduced size cylinders under conditions which could be reproduced with the large tooling.

## 1. Rod Extrusions

## a) <u>Billet Design</u>

In the work aimed at producing cups as well as in previous rod extrusions it was shown that aluminum cans will lead to irregular metal flow of the high strength metal powders. In the present work the aluminum can was replaced by a copper can. Copper has two advantages, it is considerably stiffer than aluminum at the proposed extrusion temperature, and it can be removed chemically from the composites. (The magnesium rich composite may be attacked by nitric acid which is used to remove the copper.)

The cans for the rod extrusions consisted of copper tubing, 3-inch 0.D.  $\times$  0.109-inch wall  $\times$  6-1/2 inch long. The tube was closed at both ends by a copper end plate welded to the can. One of the end plates was provided with an evacuation tube. The thickness of the end plate was originally 1/4 inch.

## b) Extrusion

All billets were filled with 70<sup>V</sup>/oAl-30<sup>V</sup>/oMg alloy powders. Table I gives details of the extrusions carried out. A total of six extrusions were attempted. Of these, two billets stalled due to excessive pressure. All billets were extruded into a water filled quench tube attached to the press.

Extrusion #5099-1, heated for 10 minutes in boiling water, was extruded successfully at a reduction ratio of 6:1. It appeared that the extrusion force was excessively high and in particular, the breakthrough pressure, probably produced by the copper end plate was close to stalling. An attempt to extrude a second billet with a reduction ratio of 10:1 resulted in a stall. An attempt was then made to thin out the front copper plate to 1/16 inch (Billet #5099-3). This also resulted in a stalled extrusion. It was obvious that an extrusion at a reduction ratio of 16:1 was not feasible at an extrusion temperature of 212°F.

In order to lower the breakthrough and running pressure, it was felt that the copper in the can and end plug should be annealed. (The copper used in the first three billets was cold worked and would not anneal at the extrusion temperature.) This idea was tested by extruding a solid copper 3-inch diameter billet, which had been annealed at 1250°F for 2 hours at a reduction in area of 10%. This extrusion (Billet #5104-1 was heated in boiling water and extruded successfully with a peak force of 675 tons and a running force of 600 tons.

Another copper can was prepared and annealed at  $1200^{\circ}F$  for 2 hours. This can was filled with  $70^{V}/oAl-30^{V}/oMg$  powder and was successfully extruded from boiling water at a reduction in area of 10:1. Peak extrusion force was 675 tons, running force 480 tons (Billet #5109-1).

At the request of the technical program manager, the two formerly stalled billets were extruded at a temperature of 425°F (heated in an electric furnace for 1 hour) at reductions of 6:1 and 10:1. At this temperature extrusion forces were low (Billets #5126-1 and 5126-2).

### c) Evaluation

All copper canned extruded rods appeared to be generally sound and uniform in cross section. The copper canning was continuous and uniform with only minor breaks in the low temperature extrusions.

Some copper breakage occurred in the 450°F extrusions, probably due to the relative stiffness of the copper compared to the Al/Mg mixtures at these temperatures. The rods were transferred to AMMRC after specimen for metallography and compressive tests had been taken from the center of the rods.

Figures 9 through 12 show longitudinal cross sections at 25% of the four successfully extruded rods. Figures 9 and 10 show the rods extruded at 212°F. The magnesium fibers are considerably more elongated in the 10:1 extrusion (Figure 10) than in the 6:1 extrusion (Figure 9). The uniformity of particle dispersion is good and relatively few particles of magnesium cluster together. Figure 11 and 12 show the rods extruded at 450°F. At a magnification of 25% the fibering at 425°F appears to be essentially similar to that observed at 212°F.

Two compression test specimens, 1/2-inch 0.D., 1-1/2 inches long were machined from each of four extruded rods. One specimen from each rod was aged at 250°F for 24 hours. The specimens were tested in a 60,000-pound Tinius Olsen testing machine at a head travel speed of 0.02 inch/minute.

In all cases the test resulted in a smooth curve with no real yield point and a steadily increasing load to the point of failure. The data reported in Table II represents the ultimate stress reached just before failure. Failure was a typical 45° shear break leaving a smooth uniform shear surface across the specimen.

The results indicate that all the extruded rods have considerable strength and that there was no aging response. In all cases the aged specimens are slightly weaker than the as-extruded and quenched specimen. Density measurements were taken on two specimens from each extruded bar. The results are shown in Table III. The theoretical density of a  $70^{\text{V}}/\text{o}$  7075,  $30^{\text{V}}/\text{o}$  ZK60A mixture is 2.509 g/cm<sup>3</sup>. Metallographically, all extruded rods showed no voids and 100% density. Therefore, one may conclude that the differences in density shown in Table III must be due to differences in distribution of the magnesium within the aluminum matrix. (The density of the 7075 alloy is 2.80 g/cm<sup>3</sup> and that of the ZK60A alloy is 1.83 g/cm<sup>3</sup>.)

A more extensive evaluation of physical properties is expected to be carried out by AMMRC on material transferred to AMMRC.

#### 2. Tube Extrusions

#### a) Billet Preparation

Three copper extrusion billets were prepared. The billets had an outer can 4-1/2 inch 0.D. x 0.134-inch wall, an inner can 1-3/8 inch 0.D. x 0.065-inch wall. The cans were 6-inches long. End plates were 1/8-inch thick. The cans were annealed at 1200°F for two hours. One end plate was welded into the can, the powder mixtures  $(70^{\text{V}}/\text{o}\ 7075-30^{\text{V}}/\text{o}\ ZK60A})$  were poured into the can and the outer end was welded. The can was then evacuated and sealed.

## b) Extrusion

The extrusion data is given in Table IV. All billets were heated in boiling water (212°F) for 15 minutes. The extrusions were carried out in a 4.555-inch liner, with a 1.750-inch die and a 1-inch mandrel resulting in a reduction ratio of 9.5:1. The first tube was extruded successfully at a ram speed of 18 inches/minute. A small amount of surface tearing occurred. In the second extrusion, the speed was lowered to 15 inches/minute. The amount of surface tearing was not reduced appreciably. A third billet was extruded at 60 inches/minute. This high speed resulted in overheating of the alloy and the extrusion was expelled from the press with considerable violence and was broken in many fragments. The two first extrusions were water quenched immediately after extrusion.

#### c) Evaluation

A sample for metallography was taken from the center of extrusion #5124-1. Normal fibering similar to that found in corresponding rod extrusions can be seen in Figure 13.

Density, compression strength, tensile strength and elongation of tubular extrusion #5124-1 in the as-extruded condition is shown in Table V. The properties of the tubes appear to be similar to those of solid rod extrusions.

## V. CONCLUSIONS AND RECOMMENDATIONS

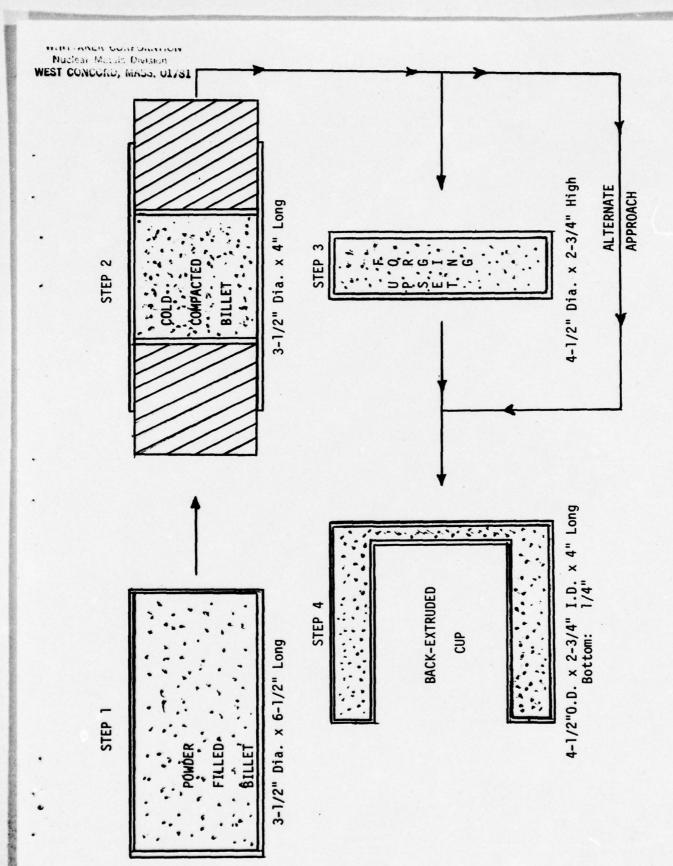
Two different processes for the production of large pipes from mixtures of microquenched spherical 7075 aluminum and ZK60A magnesium alloys have been explored. The first process consists of a consolidation, upset forging and back extrusion (cupping) sequence. The second process consists of a direct extrusion of the loose powders to the final shape.

As a result of the work reported here, it appears that only the direct extrusion approach is feasible.

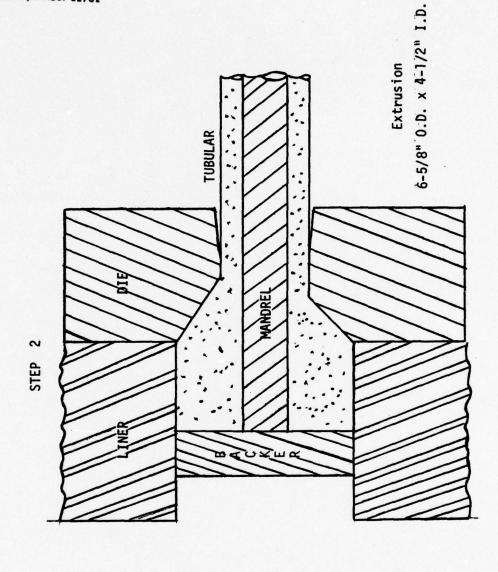
The direct extrusion produced sound rods at a reduction in area of 6:1 and 10:1. In both cases, extrusion pressures were extremely high. Therefore, large scale work should be limited to the lower reduction ratio. As part of this work, copper canning was used instead of the aluminum cans used in p-evious programs. It was found that this yields sound rods with uniform cross sections. The copper should be annealed in order to lower extrusion pressures. An extrusion temperature of 212°F (boiling water) appears to represent a practical compromise between the requirement for lower temperatues for enhanced physical properties and higher temperatures which would facilitate the extrusion process.

Small scale tubular extrusion were produced successfully, using annealed copper cans, a temperature of 212°F and a reduction ratio of 9.5:1. The data obtained in these extrusions have permitted the design of an extrusion process capable of yielding 6-inch 0.D. x 1/2-inch wall cylinders which will be required in the eventual application for AMMRC.

The apparent lack of response to heat treatment of the 7075 alloy still presents a problem. This may be caused by conditions inherent in the production of 7075 microquenched powder or by the fabrication steps. A more basic investigation of this problem is recommended.



FLOW STEP SHEET FOR GENERATION OF BACK-EXTRUDED TUBES. (Note: Dimensions are overall dimensions including canning.) FIGURE 1.



12-1/2" 0.D. x 4-3/4" I.D.

FILLED BILLET

STEP 1

Figure 2. FLOW STEP SHEET FOR DIRECT EXTRUSION OF TUBING. (Note: Dimensions are overall dimensions including canning.)

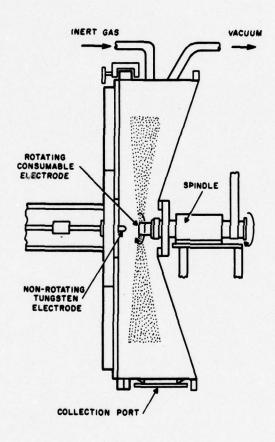


Figure 3. Schematic of The Rotating Electrode Process (REPTM) Patent #3,099,041.

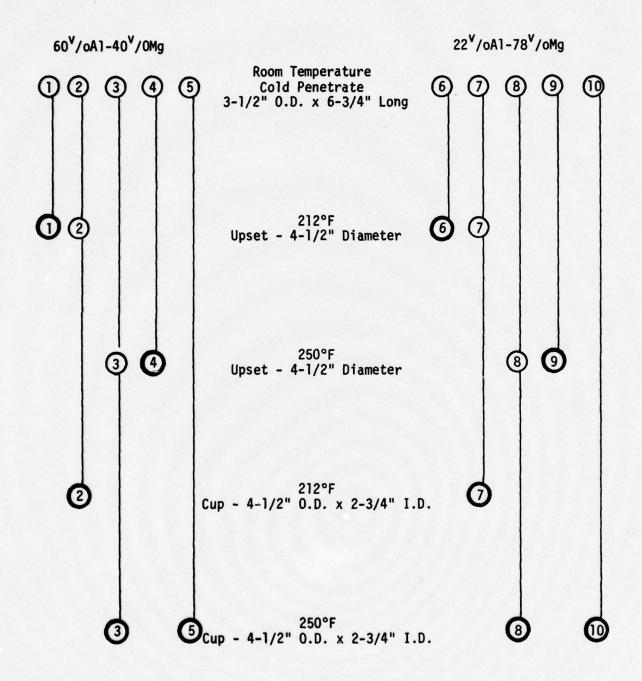


Figure 4. Flow Chart for Pancakes and Cups. 7075/ZK60A Powder Mixtures

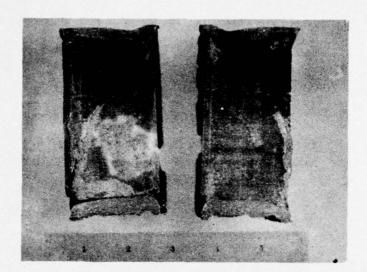


Plate #169A

Figure 5. Billet #4;  $60^{V}/oAl - 30^{V}/oMg - Cold$  Compacted at Room Temperature and Warm Upset at 250°F.

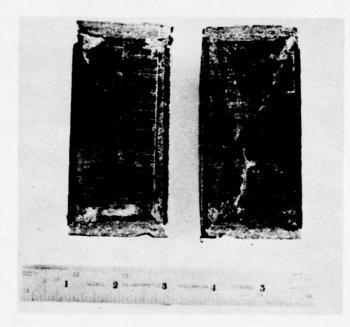


Plate #169B

Figure 6. Billet #9; 22<sup>V</sup>/oAl - 78<sup>V</sup>/oMg — Cold Compacted at Room Temperature and Warm Upset at 250°F.

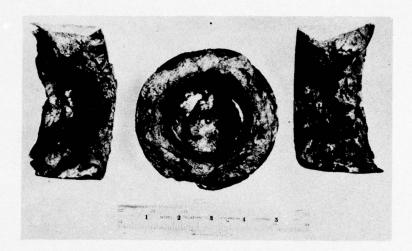


Plate #169

Figure 7. Billet #3;  $60^{\text{V}}/\text{oAl} - 40^{\text{V}}/\text{oMg} - \text{Cold Compacted at Room Temperature, Warm Upset at 250°F, and Back-Extruded at 250°F.}$ 

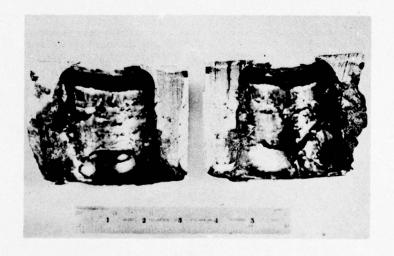


Plate #1690

Figure 8. Billet #10; 22<sup>V</sup>/oA1 - 78<sup>V</sup>/oMg — Cold Compacted at Room Temperature and Back-Extruded at 250°F. Billet was not upset forged.



Plate #400C

25X

Figure 9.  $70^{V}/o$  7075 -  $30^{V}/o$  ZK60A Extruded at 212°F.

R = 6:1 Longitudinal



Plate #400B

25X

Figure 10.  $70^{\text{V}}/\text{o}$  7075 -  $30^{\text{V}}/\text{o}$  ZK60A Extruded at 212°F. R = 10:1 Longitudinal

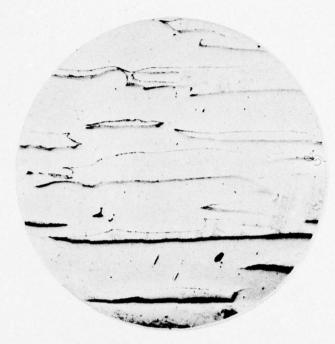


Plate #400

25X

Figure 11.  $70^{\text{V}}/\text{o}$  7075 -  $30^{\text{V}}/\text{o}$  ZK60A Extruded at 425°F. R = 6:1 Longitudinal



Plate #400A

25X

Figure 12.  $70^{\text{V}}/\text{o}$  7075 -  $30^{\text{V}}/\text{o}$  ZK60A Extruded at 425°F. R = 10:1 Longitudinal

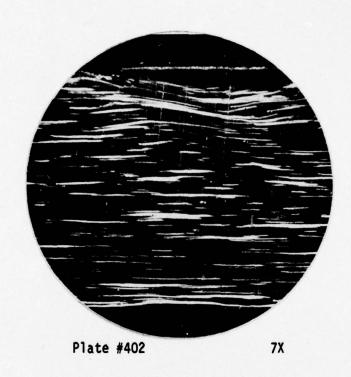




Figure 13. 70<sup>V</sup>/o 7075 - 30<sup>V</sup>/o ZK60A Tubular Extrusion at 212°F, 9.5X. Longitudinal

TABLE I

EXTRUSION OF SOLID RODS  $70^{4}/o$  7075  $-30^{4}/o$  ZK60A

Extrusion	Extrusion Temperature	Extrusion Die Opening	5	Extrusion Force (tons)	tons)	Extrusion Constant (tsi)	Extrusion Constant (tsi)	End Seal Thickness	Remarks
<u>.</u>	(°F)	(in.)	Ratio	Upset	Ruming	Upset	Upset Running	(in.)	
1-6605	212	1.225	<b>8</b>	365	465	43.2	43.2 35.5	1/4	Cold worked copper.
2-6609	212	0.950	ğ	Stall ~730 tons	coms	Stall ~43 toms	Stall 13 tons	1/4	Cold worked copper.
5099-3	212	0,950	<b>10</b>	Stall -730 tons	E J	Stall >43 toms	Stall 13 tons	1/16	Cold worked copper.
1-6015	212	0.950	ğ	675	8	\$	28.5	1/1	Annealed copper.
5126-1	425	1.225	8	365	275	8	36	*	From stalled billet 5099-2.
5126-2	425	0.950	ğ	420	350	82	12	1/4	From stalled billet 5099-3.
1-1015	212	0.950	ğ	675	009	9	35.6		Annealed solid copper.
									The state of the s

Liner Size: 3.045 inch. Copper Can: 3-inch 0.D. x 0.109-inch well. Extrusion Speed: 30-inches/minute.

TABLE IÍ

COMPRESSIVE STRENGTH OF "AS-EXTRUDED" AND AGED 70 7075 - 30 70 ZK60A POWDER MIXTURES

Extrusion	Extrusion	Reduction	Compressiv	e Strength
No.	Temperature	Ratio	As-Extruded	Aged*
5099-1	212°F	6:1	85,200 psi	81,900 psi
5109-1	212°F	9:1	85,700 psi	79,600 psi
5126-1	425°F	6:1	71,700 psi	70,900 psi
5126-2	425°F	9:1	81,400 ps1	80,600 psi

<sup>\*</sup> Aged for 24 hours at 250°F.

TABLE III

DENSITY OF EXTRUDED 70 0 7075 = 30 1/6 ZK60A MIXTURES

Extrusion	Extrusion	Reduction	Di	ensity		
No.	Temperature	Ratio	g/cm <sup>3</sup>	Theoretical*		
5099-1	212°F	6:1	2.643 2.589	105.3% 103.4%		
5109-1	212°F	9:1	2.644 2.630	105.4%		
5126-1	425°F	6:1	2.517 2.400	100.3% 95.6%		
5126-2	425°F	9:1	2.543 2.587	101.3% 103.1%		

<sup>\*</sup>Theoretical Density: 2.509

TABLE IV EXTRUSION OF TUBES

70<sup>V</sup>/o 7075 - 30<sup>V</sup>/o ZK60A

Extrusion	Extrusion Speed	Extrusi (to	Extrusion Force (tons	Extrusion (1	Extrusion Constant (tsi)
No.	(ipm)	Upset	Running	Upset	Running
5124-1	18	1275	975	36.5	29.0
5124-2	51	1330	950	38.1	27.2
5124-3	09	1275	950	36.5	27.2

4.555-inch Liner -1.75-inch Die -1-inch Mandrel -R = 9.5:1 Billets Heated to 212°F.

TABLE V

PHYSICAL PROPERTIES OF TUBULAR
EXTRUSION # 5124-1 (As-Extruded)

	SAMPLE A	SAMPLE B
Density	2.536 g/cm <sup>3</sup> 101.25% of theoretical	2.543 g/cm <sup>3</sup> 101.51% of theoretical
Compressive Strength	81,997 psi	81,895 psi
Tensile Strength	53,297 psi	55,256 psi
Tensile Elongation in 1/2 inch Gage Length	5.6%	6.7%

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